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NASA Case No. LEW-15,263-1

PRINT FIG. 1

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Serial No.: 892,054

Filed Date: 6/4/92

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LeRC

EMPLOYER : NASA Lewis Research Ctr.

(NASA-Case-LEW-15263-1) SILICON
CARBIDE FIBER REINFORCED STRONTIUM
ALUMINOSILICATE GLASS-CERAMIC
MATRIX COMPOSITE Patent Application
(NASA) 7 p

N93-11543

Unclass

Silicon Carbide Fiber Reinforced Strontium
Aluminosilicate Glass-Ceramic Matrix Composite

5 Origin of the Invention

The invention described herein was made by an employee of the U.S. Government and may be manufactured or used by or for the Government without the payment of any royalties thereon or therefor.

10 Technical Field

This invention is concerned with a ceramic fiber-reinforced glass-ceramic matrix composite. The invention is particularly concerned with a high strength ceramic fiber-reinforced composite which is refractory, strong, and tough
15 for applications as high temperature structural materials.

It has been suggested that lithium aluminosilicate, magnesium aluminosilicate (cordierite), and calcium aluminosilicate glass ceramics reinforced with SiC fibers derived from polymers be used for such applications. Commercially
20 available SiC fibers, known as Nicalon, have been satisfactory. However, the potential use of such prior art materials is limited to somewhat low temperatures of about 1000°C to 1100° C.

The fabrication of a composite by infiltration of melt of
25 SrO-Al₂O₃ - SiO₂ (SAS) composition into a preform made out of SiC powder, SiC whiskers and CVD SiC fibers has been attempted. However, the fiber-reinforced composites made by this method showed no improvement in flexural strengths over the ones with no reinforcements.

30 It is, therefore, an object of the present invention to provide a new composite material having superior mechanical properties at high temperatures.

Another object of the invention is to provide a method of

making a ceramic fiber-reinforced glass-ceramic matrix composite material for use in gas turbine and diesel engines.

Background Art

5 Hillig patent No. 4,725,567 is directed to a composite material processed by infiltration. The object of the invention is the production of a high temperature composite material. Hillig patent No. 4,788,162 discloses a method for the production of high temperature composite materials. This
10 patent notes that the SrSiO_3 has unique characteristics, such as expansion while freezing. The combination of CaSiO_3 and SrSiO_3 provides a material which is stable at a variety of temperature ranges.

15 Hillig and McGuigan patent No. 4,917,941 is directed to a ceramic matrix produced with continuous SiC fibers and a slip casting technique. A porous mold in the shape of the material desired is used in the process. It is capable of removing the liquid from the suspension leaving only the matrix material. Prior to drying, continuous SiC fibers are wrapped around the
20 mold. The process may be repeated to add thickness to the material. Drying occurs in an ambient environment.

Gadkaree patent No. 4,919,991 is concerned with another composite. The only pertinency of this patent is the use of SrSiO_3 as an alternative material in the composite structure.
25 Singh et al patent No. 4,944,904 discloses a ceramic matrix having a boron nitride coating. A carbon cloth is dipped into a slurry layer into a preform. At this point infiltration is used to form the composite.

Disclosure of the Invention

30 The objects of the invention are achieved with a fiber-reinforced composite which is composed of a $\text{SrO-Al}_2\text{O}_3-2\text{SiO}_2$ (SAS) glass ceramic matrix that has been reinforced with CVD SiC continuous fibers. The method of assembly includes

preparing a slurry of SAS glass powders and casting this
slurry into tapes. After the tapes are dried they are cut to
the proper size.

Continuous CVD-SiC fibers are formed into mats of the
desired size. The matrix tapes and the fiber mats are
alternately stacked in the proper orientation. This tape-mat
stack is warm pressed to produce a "green" composite.

The "green" composite is then heated to an elevated
temperature to burn out organic constituents. The remaining
interim material is then hot pressed to form a SAS glass-
ceramic fiber-reinforced composite which may be machined to
size.

Description of the Drawing

The objects, advantages and novel features of the
invention will be more fully apparent from the following
detailed description when read in conjunction with the
accompanying drawing which is a graph showing three point
flexural stress-strain behavior of a 24 volume percent silicon
carbide fiber-reinforced SAS glass-ceramic composite material
at room temperature.

Best Mode for Carrying Out the Invention

The composite of the present invention is produced by a
tape method in which an aqueous slurry of SAS glass powder
along with a fugitive organic binder, plasticizer, glycerine,
and surfactant is prepared. Glass powders having an average
particle size of about 2.5 μm have been satisfactory. A
binder identified as Methocel 20-214 which is a commercially
available material from the Dow Chemical Company has been
suitable. A plasticizer known commercially as Polyglycol E-
400 from the Dow Chemical Company has been adequate. A
surfactant material sold commercially as Tritonex 100 has
provided satisfactory results.

This slurry is first ball milled and then cast into tapes. This may be facilitated by using a doctor blade. After the tape has dried, it is cut to size. The composition of the matrix is preferably about 31.8 w/o SrO, about 31.3 w/o Al₂O₃, and about 36.9 w/o SiO₂. This composition is also expressed as 25 mole % SrO, 25 Mole % Al₂O₃, and 50 mole% SiO₂.

Continuous CVD SiC fibers that were supplied by Textron Specialty Materials are wound onto a drum. Commercially available adhesive tape is used to hold the fibers in their proper position on the drum. The fibers are then cut into unidirectional mats of desired predetermined size whose fiber orientation and integrity are maintained by the adhesive tape.

The required number of matrix tapes and fiber mats are alternately stacked in a desired orientation in a warm press die. After stacking, this material is warm pressed to produce a "green" composite.

The "green" composite is then removed from the warm press die and wrapped in either graphite foil or molybdenum foil. The wrapped "green" composite is loaded into a hot pressing die where it is heated to an elevated temperature between about 400° C to 500° C to burn out organic constituents to produce an interim material.

The interim material is then hot pressed under vacuum using temperatures of 1200° C - 1500° C, a pressure of 2 to 4 KSI, and a time of 15 to 120 minutes. After cooling to room temperature and pressures, the resulting composites are then removed from the die.

A number of the composites were cut into test bars for flexural strength measurements and other characterizations. Referring to the drawing there is shown a graph of the 3-point flexural stress-strain behavior for a 24 volume percent SiC fiber/SAS glass-ceramic composite material. Unidirectional

composites having room temperature 3-point flexural stress of greater than 1000 MPa have been fabricated with 27 volume percent fiber loading.

5 While the preferred embodiment of the invention has been shown and described, it will be appreciated that various modifications may be made to the invention without departing from the spirit thereof or the scope of the subjoined claims. This silicon carbide fiber-reinforced SAS glass-ceramic matrix
10 composite is a new and improved structural material. The composite fabricated by the aforementioned method has mechanical properties superior to existing materials and a potential for use at temperatures as high as 1450°C to 1500°C.

Abstract of the Disclosure

SILICON CARBIDE FIBER REINFORCED STRONTIUM
ALUMINOSILICATE GLASS-CERAMIC MATRIX COMPOSITE

A $\text{SrO-Al}_2\text{O}_3 - 2\text{SrO}_2$ (SAS) glass ceramic matrix is reinforced with CVD SiC continuous fibers. This material is prepared by casting a slurry of SAS glass powder into tapes. Mats of continuous CVD-SiC fibers are alternately stacked with the matrix tapes.

This tape-mat stack is warm-pressed to produce a "green" composite. Organic constituents are burned out of the "green" composite, and the remaining interim material is hot pressed.

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